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The effects of heat treatment on physical and technological properties and surface roughness of Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood

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Abstract

Heat treatment is often used to improve the dimensional stability of wood. In this study, the effects of heat treatment on physical properties and surface roughness of Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood were examined. Samples obtained from Yenice-Zonguldak Forest Enterprises, Turkey, were subjected to heat treatment at varying temperatures and for varying durations. The physical properties of heat-treated and control samples were tested, and oven-dry density, air-dry density, and swelling properties were determined. The mechanical properties of heat-treated and control samples were tested, and compression strength, and Janka-hardness were determined. A stylus method was employed to evaluate the surface characteristics of the samples. Roughness measurements by the stylus method were made in the direction perpendicular to the fiber. Four main roughness parameters, mean arithmetic deviation of profile (R_a), mean peak-to-valley height (R_z), root mean square roughness (R_q), and maximum roughness (R_y) obtained from the surface of wood were used to evaluate the effect of heat treatment on the surface characteristics of the specimens. Significant difference was determined ($p = 0.05$) between physical and technological properties, and surface roughness parameters (R_a, R_z, R_y, R_q) for three temperatures and three durations of heat treatment.

Based on the findings in this study, the results showed that density, swelling, compression strength, Janka-hardness and surface roughness values decreased with increasing treatment temperature and treatment times. Increase in temperature and duration further diminished technological strength values of the wood specimens. Camiyanı Black Pine wood could be utilized by using proper heat treatment techniques without any losses in strength values in areas where working, stability, and surface smoothness, such as in window frames, are important factors.

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Keywords: Camiyanı Black Pine; *Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*; Physical properties; Technological properties; Surface roughness; Heat treatment

1. Introduction

Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) grows naturally in Yenice Region Turkey. It is determined that the heartwood ratio for Camiyanı Black Pine is more than 50% in comparison to the sapwood in Yenice region. Camiyanı Black Pine grows naturally in Elekdag, Dursunbey, Tosya, Daday, Tavsanli and Yenice regions of Turkey with the total area of 30,000 ha. The average habitat altitude is 866 m and prefers calcareous with stone and sandy-clay soils. It is a primary forest tree species with an average of 30 m height; rarely reaching 50 m (Berkel, 1970; Gündüz, 1999).

This species is preferred in the forest industry because of its relatively large and distinct heartwood in comparison with other pine species. The reason for that is the color

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of the heartwood darkens with time and becomes more decorative. Camiyanı Black Pine was in the endangered species list in 1986 by the government. Camiyanı Black Pine has been used for utility poles, veneer, fences, railroad ties, building foundations and beams, marine applications, bridges and trusses, and boardwalks (Gündüz, 1999).

Heat treatment is one of the processes used to modify the properties of wood (Mazela et al., 2004). Heat treatment, as a wood modification method, serves to improve the natural quality properties of the wood, such as dimensional stability and resistance to bio-corrosion and equip the wood material with new properties.

Industrial-scale heat treatment process was developed at the technical research center of Finland in the early 1990s. The total production capacity of heat-treated wood in 2002 is estimated approximately to be 265,000 m^3 (Syrjanen and Oy, 2001; Rapp, 2001). Recent efforts on thermal treatment of wood have led to the development of several treatment processes, and materials produced through thermal treatments have been introduced to the European market. Some of the products developed by thermal treatment include thermowood (Stellac) in Finland (Viitaniemi et al., 1994), torrefaction (perdure) in France (Weiland and Guyonnet, 1997) and PLATO-wood in The Netherlands (Militz, 2002).

Wood treated at high temperature has less hygroscopicity than natural wood. Heating wood permanently changes several of its chemical and physical properties. The change in properties is mainly caused by thermic degrading of hemicelluloses. Theoretically, the available OH groups in hemicellulose have the most significant effect on the physical properties of wood. Heat treatment slows water uptake and wood cell wall absorbs less water because of the decrease of the amount of wood's hydroxyl groups. As a consequence of the reduced number of hydroxyl groups the swelling and shrinking are lower. In addition to better durability the advantages of heat-treated wood are reduced hygroscopicity and improved dimensional stability. It stabilises around 4–5% in humidity instead of 10–12% (Inoue et al., 1993).

Heat treatment significantly reduces the tangential and radial swelling. The wood's swelling and shrinkage is very low. Desired changes start to appear already at about 150 °C, and the changes continue as the temperature is increased in stages (Anonymous, 2003).

In a study (Stamm and Hansen, 1937), the hygroscopicity of black gum wood decreased to half of its original value when samples were treated at 205 °C for 6 h. In another study (Yıldız, 2002), increases in temperature and treatment time, and also the technique used, resulted in changes in dimensional stability from 55% to 90%. When young beech, oak, and pine wood was heat-treated at 70–200 °C between 6 h and 48 h, the effect of heat treatment was obvious after 100 °C, and sorption capacity decreased with increased treatment time and temperature (Kollmann and Schneider, 1963). In addition, heat treatment resulted in varying amounts of weight loss, depending on the treatment temperature and time. For spruce (*Picea abies*) wood, a 24 h heat treatment resulted in 0.8% and 15.5% weight loss at 120 °C and 200 °C, respectively. Weight loss of beech (*Fagus sylvatica*) wood, treated at increasing temperatures, was 8.1% and 9.8% at 150 °C and 200 °C, respectively (Fengel and Wegener, 1989).

Heat treatment reduces certain mechanical properties, but the dimensional stability and the biological durability of wood increases through heat treatment. Also, heat treatment results in favorable changes in the physical properties of the wood, such as reduced shrinkage and swelling, low equilibrium moisture content, enhanced weather resistance, a decorative dark color, and better decay resistance (Yıldız et al., 2006). Therefore, heat-treated wood is an eco-friendly alternative to impregnated wood materials, and heat-treated wood can be used for garden, kitchen and sauna furniture, cladding on wooden buildings, bathroom cabinets, floor material, musical instruments, ceilings, inner and outer bricks, doors and window joinery, and a variety of other outdoor and indoor wood applications (Syrjanen and Oy, 2001).

Research on the effects of heat treatment on the physical and mechanical properties and surface roughness of Turkish native trees is rather limited. This study aims at examining the effects of heat treatment on physical and mechanical properties and surface roughness of Camiyanı Black Pine (*P. nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood, which has not been done before. Camiyanı Black Pine is one of the most common wood species naturally grown and intensively used in the forest product industry in Turkey. Improving the characteristics of Camiyanı Black Pine through heat treatment would offer the timber product industry many interesting opportunities.

2. Methods

Three trees with a diameter at breast height diameter (DBH. 1.3 m above ground) of 68–88 cm were obtained from Yenice-Zonguldak forest Enterprises (TS 4176). The area from which the trees were taken was at an elevation of 1330 m and had a slope of 25–30%. Lumber from the logs was prepared by AKE Sawmill Ltd. Camiyanı Black Pine lumber was finished by a fixed-knife planer with a feed speed of 1 m/s. The bias angle of the knife was 45° for the lumber. If the wood pieces are sawn so that the annual rings are at least in 45° angle to the surface the deformations will be smaller, the hardness of the surface will be stronger and the "general looks" after heat treatment is better. Sampling and tests were performed according to several Turkish and ISO standards. Small clear samples were obtained for density and swelling (20 × 20 × 30 mm), compression strength parallel to grain (20 × 20 × 30 mm), Janka-hardness (50 × 50 × 50 mm) and surface roughness measurements (50 × 50 × 50 mm) (TS 2470).

The number of specimens taken from each log was equal. Specimens were divided into nine treatment groups and for each group, a total of 30 test and 30 control samples were used. The samples were subjected to heat treatment at 120 °C, 150 °C, or 180 °C for 2, 6, or 10 h in a small heating unit controlled to within ±1 °C under atmospheric pressure. After heat treatment, treated and untreated samples were conditioned to 12% moisture contents (MC) in a conditioning room at 20 °C ± 2 °C and with 65% (±5) relative humidity (RH) according to TS 642. The air-dry density of the samples was determined. The dimensions and weights of the samples were measured. The oven-dry and air-dry density of the samples was determined at 0.01 mm and 0.001 gr sensitivity.

After the oven-dry dimensions were determined. the samples were soaked in water (20 ± 2 °C). When no changes in sample dimensions were observed. the dimensions were measured.

Tests for density (30 samples) and swelling (30 samples), compression strength parallel to grain (30) and Janka-hardness (30) were carried out based on (ISO 3131), (TS 4084), (TS 2595), and (TS 2479) respectively.

Upon conducting the experiments the moisture contents (the moisture content deviation from 12%) of the specimens were measured according to (TS 2471) and strength values were corrected (transformed to 12% moisture content) by using the following strength conversion equation:

$$\delta_{12} = \delta_{\mathrm{m}} * [1 + \alpha(M_2 - 12)]$$

where δ_{12} = strength at 12% moisture content (N/mm^2), δ_{m} = strength at moisture content deviated from 12% (N/mm^2), α = constant value showing relationship between strength and moisture content, M_2 = moisture content during the experiment (%).

Surface roughness of the samples was measured by using a profilometer (Mitutoyo Surftest SJ-301). The surface roughness of the samples was measured with the profile method using a stylus device standard. The measuring speed, pin diameter, and pin top angle of the tool were 10 mm/min, 4 μm, and 90°, respectively. The points of roughness measurement were randomly marked on the surface of the samples. Measurements were made in the direction perpendicular to the fiber of the samples.

Three roughness parameters, mean arithmetic deviation of profile (Ra), mean peak-to-valley height (Rz), and maximum roughness (Ry) were commonly used in previous studies to evaluate surface characteristics of wood and wood composites including veneer (Stombo, 1963). Ra is the average distance from the profile to the mean line over the length of assessment. Rq is the square root of the arithmetic mean of the squares of profile deviations from the mean line. Rz can be calculated from the peak-to-valley values of five equal lengths within the profile while maximum roughness (Ry) is the distance between peak and valley points of the profile which can be used as an indicator of the maximum defect height within the assessed profile (Mummery, 1993). Therefore, such parameters which are characterized by (ISO 4287) and (DIN 4768) were recorded.

Specification of this parameter is described by Hiziroglu (1996) and Hiziroglu and Graham (1998). Roughness values were measured with a sensitivity of 0.5 μm. The length of scanning line (*L*t) was 15 mm and the cutoff was $\lambda = 2.5$ mm. The measuring force of the scanning arm on the surfaces was 4 mN (0.4 g), which did not put any significant damage on the surface according to Mitutoyo Surftest SJ-301 user manual (Anonymous, 2002). Measurements were performed at room temperature and the pin was calibrated before the tests. Fig. 1 shows the Mitutoyo Surftest SJ-301.

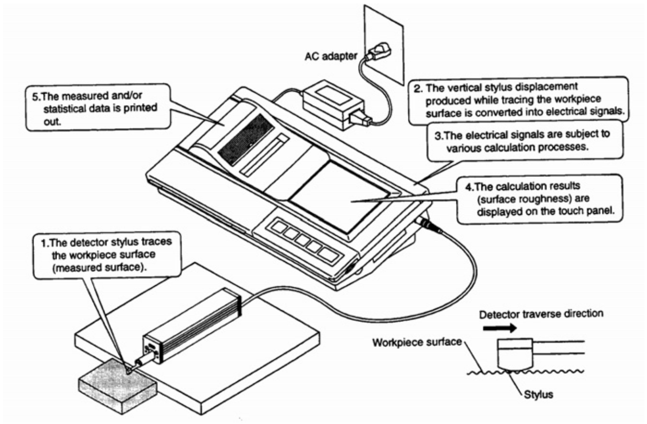
AC adapter
5.The measured and/or statistical data is printed out.
2. The vertical stylus displacement produced while tracing the workpiece surface is converted into electrical signals.
3.The electrical signals are subject to various calculation processes.
4.The calculation results (surface roughness) are displayed on the touch panel.
1.The detector stylus traces the workpiece surface (measured surface).
Detector traverse direction
Workpiece surface
Stylus

Fig. 1. Schematic description of the Mitutoyo Surftest SJ-301.

For the oven-dry density, air-dry density, swelling, compression strength parallel to grain, Janka-hardness and average roughness, all multiple comparisons were first subjected to an analysis of variance (ANOVA) and significant differences between mean values of control and treated samples were determined using Duncan's multiple range test.

3. Results and discussion

Table 1 shows the oven-dry and air-dry densities and swelling ratios under the different treatment regimes. Table 3 shows the results of compression strength parallel to grain and Janka-hardness, for the control and all the different heat treatment and time combinations. According to the averages, all the parameters decreased with increasing temperature and time. It is evident from the results that these values were all lower in heat-treated samples than in control samples. The effect of the heat treatments was significant for all the variables analyzed.

It is clear from this study that the value of all measured physical and technological properties and surface roughness decreased with increasing temperature and duration. Tables 2 and 4 shows the percentage decrease of values in relation to the control for each treatment and each measured parameter.

It is evident from Table 1 that the oven-dry density and air-dry density values decrease with increasing temperature and heat treatment time under the conditions used. Heat-treated wood samples at a temperature of 180 °C for 10 h gave the lowest air-dry and oven-dry density values when compared with other conditions studied.

Table 1
The effect of heat treatment for different durations on physical properties and surface roughness in Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood[a]

Heat treatment	Time (h)	Unit[b]	Oven-dry density[c] Do (g/cm^3)^3	Air-dry density[c] D_{12} (g/cm^3)	Surface roughness[c] Ra (μm)	Rz (μm)	Ry (μm)	Rq (μm)	Swelling[c] Radial (%)	Tangential (%)	Longitudinal (%)
Control		Mean	0.540 A	0.599 A	8.025 A	53.622 A	73.485 A	10.471 A	4.177 AJ	7.605 A	0.991 ABCD
		±sd	0.085	0.095	2.362	13.865	16.811	2.739	1.195	1.258	0.591
120 °C	2	Mean	0.538 A	0.574 A	7.750 AEFGHI	51.614 AEF	73.331 AB	9.861 AF	4.025 BJ	6.074 BIJ	0.978 ABCD
		±sd	0.063	0.130	1.200	9.886	17.381	2.647	1.191	1.348	0.477
	6	Mean	0.536 A	0.565 A	7.235 B	49.586 A	69.186 AB	9.511 A	3.971 CJ	5.904 CJ	0.950 ABCD
		± sd	0.061	0.059	0.732	8.087	2.457	23.177	1.168	1.149	0.691
	10	Mean	0.533 A	0.546 A	7.177 C	49.071 A	66.657 A	9.458 A	3.906 D	5.878 DJ	0.934 ABCD
		±sd	0.057	0.127	1.013	7.2567	12.443	1.514	1.459	1.685	0.537
150 °C	2	Mean	0.532 A	0.543 A	7.043 D	48.860 A	65.513 A	9.091 A	3.837 E	5.358 E	0.914 ABCD
		±sd	0.092	0.066	1.192	8.271	18.337	3.142	1.395	1.068	0.479
	6	Mean	0.530 A	0.536 A	6.968 E	48.015 B	64.813 A	8.831 B	3.789 F	5.275 F	0.904 ABCD
		±sd	0.066	0.073	1.083	9.371	16.850	2.2614	1.046	1.299	0.377
	10	Mean	0.523 A	0.535 A	6.902 F	47.726 C	64.264 A	8.802 C	3.573 G	5.063 G	0.866 BD
		±sd	0.057	0.053	1.539	10.773	16.626	2.197	1.111	1.694	0.445
180 °C	2	Mean	0.520 A	0.528 A	6.896 G	47.398 D	63.848 A	8.749 D	3.565 H	4.994 H	0.828 BD
		±sd	0.072	0.052	1.483	9.005	16.059	2.908	1.455	1.290	0.378
	6	Mean	0.512 B	0.516 B	6.833 H	45.938 E	63.358 A	8.720 E	3.503 I	4.719 I	0.754 CD
		±sd	0.048	0.152	1.175	8.813	17.023	2.061	1.211	1.309	0.361
	10	Mean	0.506 C	0.509 C	6.765 I	45.811 F	58.866 B	8.096 F	3.262 J	4.698 J	0.719 D
		±sd	0.069	0.112	1.056	6.867	14.845	2.572	0.944	1.265	0.413

[a] Number of samples used in each test is 30.

[b] Avg. = average; sd = standard deviation

[c] Homogenous groups: letters in each column indicate groups that are statistically different according to Duncan's multiple range test at $P < 0.05$. Comparisons were between each control and its test.

Table 2
Percentage decrease of physical properties and surface roughness in Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood following heat treatment for different durations

Heat treatment (°C)	Time	Oven-dry density (%)	Air-dry density (%)	Swelling Radial (%)	Tangential (%)	Longitudinal (%)	Surface roughness Ra (%)	Rz (%)	Ry (%)	Rq (%)
120	2	22.163	4.151	19.126	37.613	1.274	3.423	3.744	0.209	5.822
	6	23.073	5.534	20.201	39.359	4.137	9.844	7.526	5.85	9.171
	10	26.122	8.736	21.509	39.618	5.752	10.57	8.486	9.292	9.678
150	2	26.148	9.203	22.904	44.961	7.770	12.23	8.881	10.85	13.18
	6	26.358	10.397	23.855	45.815	8.779	13.18	10.46	11.8	15.66
	10	27.416	10.646	28.211	47.996	12.610	14	10.99	12.55	15.94
180	2	27.863	11.812	28.367	48.706	16.370	14.07	11.61	13.11	16.45
	6	28.914	13.832	29.607	51.521	23.850	14.85	14.33	13.78	16.73
	10	**29.746**	**14.894**	**34.460**	**51.738**	**27.360**	**15.71**	**14.57**	**19.89**	**22.68**

Decreases in swelling to radial, tangential and longitudinal directions were found to be 34.460%, 51.738%, and 27.360%, respectively, when treated at 180 °C for 10 h. A decrease in swelling results in an increase in dimensional stability, which is required for several uses of wood.

Surface roughness decreased by up to 15.71% in the sample heat-treated at 180 °C for 10 h when compared with the control samples. This increase in smoothness is very important for many applications of solid wood. In addition, losses occurring in the planing machine are reduced and high quality surfaces are attained.

The maximum decreases for all parameters were recorded at the treatment of 180 °C for 10 h. The lowest compression strength values obtained was 41.432 N/mm^2, total loss compared to the control was calculated to be 27.2%. Maximum hardness loss was obtained for samples

Table 3
The effect of heat treatment for different durations on compression strength and Janka-hardness in Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood[a]

Heat treatment	Time (h)	Unit[b]	Compression strength[c] (N/mm^2)	Janka-hardness		
				Cross-section (N/mm^2)	Radial (N/mm^2)	Tangential (N/mm^2)
None		Avg.	56.935 A	47.58 A	24.546 A	17.713 A
		±sd	12.821	11.543	12.266	6.225
120 °C	2	Avg.	54.324 ACDEFG	45.546 ACDEFG	19.99 BIGJ	17.446 ABCDEF
		±sd	12.296	8.656	2.961	9.994
	6	Avg.	52.123 AEFG	44.27 AG	19.736 CIJ	17.15 ABCDEF
		±sd	10.561	12.271	5.831	4.198
	10	Avg.	50.146 AFG	43.756 AG	17.863 D	15.536 A
		±sd	9.054	13.891	4.086	7.648
150 °C	2	Avg.	48.453 B	42.733 BG	17.04 E	15.273 A
		±sd	10.341	4.572	5.085	4.554
	6	Avg.	46.312 C	41.373 CG	16.713 F	15.076 B
		±sd	13.806	6.121	4.586	4.140
	10	Avg.	45.345 D	40.52 DG	16.596 G	14.086 C
		±sd	14.063	3.126	2.873	2.427
180 °C	2	Avg.	43.657 E	40.44 EG	16.446 H	13.783 D
		±sd	12.511	2.201	4.359	1.653
	6	Avg.	42.876 F	39.543 FG	15.476 I	13.453 E
		±sd	12.224	7.157	4.116	1.477
	10	Avg.	41.432 G	32.17 G	14.996 J	13.423 F
		2.262	2.451	2.662		

[a] Number of samples used in each test is 30.

[b] Avg. = average; sd = standard deviation

[c] Homogenous groups: letters in each column indicate groups that are statistically different according to Duncan's multiple range test at $P < 0.05$. Comparisons were between each control and its test.

Table 4
Percentage decrease of compression strength and Janka-hardness in Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood following heat treatment for different durations

Heat treatment (°C)	Time (h)	Compression strength	Janka-hardness		
			Cross-section (%)	Radial (%)	Tangential (%)
120	2	4.59	4.27	18.56	1.51
	6	8.45	6.96	19.6	3.18
	10	11.9	8.04	27.23	12.3
150	2	14.9	10.2	30.58	13.8
	6	18.7	13	31.91	14.9
	10	20.4	14.8	32.39	20.5
180	2	23.3	15	33	22.2
	6	24.7	16.9	36.95	24
	10	**27.2**	**32.4**	**38.91**	**24.2**

treated at 180 °C for 10 h; cross-section 32.4%, radial 38.91% and tangential 24.2%. The Janka-hardness values are greatly affected by initial treatments but after that only show a gradual decrease with increasing temperatures and treatment times. In contrast the strength values do not start to show significant changes with the lower temperature treatment of 120 °C but an increase in temperature starts to have an effect.

In general the results of this study on the effect of heat treatment on Camiyanı Black Pine are compatible with the findings in literature on the effect of heat treatment on different tree species. Ünsal et al. (2003) reported that in Turkish river red gum (*Eucalyptus camaldulensis* Dehn.) wood samples the largest hardness loss was at 180 °C for 10 h treatment. The loss was 23.91% cross-sectionally, 44.20% radially, and 33.57% tangentially. Unsal and Ayrilmis (2005) also found that the maximum compression strength parallel to grain decrease in Turkish river red gum (*E. camaldulensis* Dehn.) wood samples was 19.0% at 180 °C for 10 h treatment. Korkut (in press) obtained similar compression strength results for Uludağ fir (*Abies bornmuellerinana* Mattf.) wood for the same treatment time and temperature. In this case it can be said that temperature has greater influence on strength properties than time.

In a study on the effect of heat treatment, with pine sapwood heated at 110, 130, 150 and 180 °C it was found that compression strength decreased by 5% (Schnerder, 1973). Similarly another study showed that for Eucalyptus (*Eucalyptus saligna*) wood, compression strength parallel to grain decreased at 110–155 °C for 10–160 h treatments (Vital et al., 1983).

4. Conclusion

In conclusion, it was found that the density, swelling, compression strength parallel to grain, Janka-hardness and surface roughness of the Camiyanı Black Pine decreased for all treatment conditions (temperatures and times). The smallest decrease was observed in the treatment at 120 °C for 2 h. The largest decrease found was for swelling, followed by tangential swelling and the radial Janka-hardness, when samples were heat-treated under the specific conditions of this research.

The smallest decrease was determined at the heat treatment of 120 °C for 2 h. In this research, physical properties, surface roughness and technological strength values all decreased with increasing time and temperature treatments.

The improved characteristics in swelling and surface roughness of heat treated timber have to be balanced against the decrease in strength values when evaluating the effectiveness of using this treatment. The most important property, when compared to untreated wood, is that the equilibrium moisture content of the heat-treated wood is reduced and as a consequence of this shrinkage and swelling of the wood is also reduced. Various tree species can be utilized using proper heat treatment techniques without any losses in strength values in areas where woodworking and stability are important. Wood species having no commercial value can also be utilized effectively, following heat treatment, in areas where they previously had little potential.

Due to its good weather resistance, thermowood is suited for outdoor applications such as external cladding, window frames and garden furniture. As a result of the dimensional stability the heat-treated wood gives better durability for coating.

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